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Ferroelectric domains in pyroelectric ceramics

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Abstract : The photograph of five group specimens of Lead Titanat Zirconat ceramics with a base composition $Pb(Ti_xZr_{1-x})O_3$, $x = 0.07$ sintered at 1200°C for 1–11 hour were studied. It was observed that with increasing the sintering time, grain size increased, pyroelectric coefficient decreased and fluctuation of pyroelectric coefficient in temperatures between 20°C and 70°C decreased. By SEM photographs it is observed that the PZT pyroelectric ceramic have four types of ferroelectric domains with orientation 71°, 90°, 109° and 180°. The change of pyroelectric voltage and coefficient is due to move of these domains.

Keywords : Ferroelectric domains, pyroelectric ceramics

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Different properties of pyroelectric materials have been studied in the literature [1–9]. True pyroelectric results from the temperature dependence of the spontaneous polarization of polar materials and is therefore shown by ferroelectric materials whether they are single-domain, single-crystal or poled ceramics.

The method of preparation and characterization of pyroelectric ceramics in details are available in the literature [1,9]. Applications of the pyroelectric materials are mostly in pyroelectric detectors, integrated optical modulators and infrared detectors.

Zirconate oxide ceramics have especially good pyroelectric and piezoelectric properties depending on its composition. $Pb(Ti_xZr_{1-x})O_3$ ceramics with $x = 0.07$ have pyroelectric properties. With this ceramics, infrared detectors with fast response and high sensitivity have been made [2]. The aim of this paper is to study the ferroelectric domains and the effect of orientation of these domains on the grains by SEM photographs. An experimental procedure is given in Section 2. Section 3 gives the results and discussion. Conclusions appear in Section 4.

Lead titanate zirconate oxide with a base composition $Pb(Ti_xZr_{1-x})O_3$ with $x = 0.07$ has been calcined at 1000°C for one hour. The samples are sintered at a temperature of 1200°C with heating rate 5°C/min and a cooling rate 5°C/min and a socking time held for five sets of specimens for a period of 1, 3, 7, 9 and 11 hour. Calcining and sintering process are made with a microprocessor controlled furnace model labotherm HT 04/17 [4]. The surface of five groups of specimens was polished and etched. In the chemical etching process, two factors are important. First is the kind of the etching material and the second is the time of etching. Etching process has been done with a 0.5% HF acid solution and 95% of HCl. This solution was composed with water with a rate of ten to one. After etching for 10 minutes by ultrasonic apparatus, the surface of specimen was cleaned. This process was done for removal of crashed grains which remained on surface from etching process. In the SEM

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method, electrical charges are collected on the surface, and so the photographs of the surface were destroyed. For this reason, the surface were coated with a 2 μm thick layer of gold.

With the SEM photographs, it was observed that by increasing the sintering time, the grain size increased. In other word, the grain sizes for specimens for 1 hour and 11 hours were respectively 3.97 μm and 16.29 μm. Four types of other ferroelectric domain orientations were observed in surface of grains. These orientations are 71°, 90°, 109° and 180° and shown in Figures 1–4. Figure 1 shows the domains with orientation 71°. Figure 2 shows the domains with orientation 90°.

Figure 1. Ferroelectric domain with 71° orientation.

Figure 2. Ferroelectric domain with 90° orientation.

Lower part of Figure 3 shows the domains with orientation 109°. Figure 4 shows the domains with orientation 180°.

These orientations completely agreed with results in literature [1]. The variations of pyroelectric voltage and coefficient as functions of temperature are shown in Figure 5.

Figure 3. Ferroelectric domain with 109° orientation.

Figure 4. Ferroelectric domain with 180° orientation.

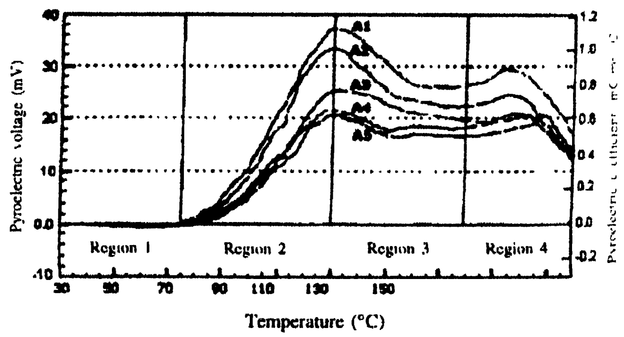

Figure 5. Variation of pyroelectric voltage and coefficient with temperature.

There are four distinguishable regions in this figure. In region 1 (between 30° and 75°), 71° and 109° domains that are unstable with respect to 90° and 180° domains reorient; then the values of pyroelectric voltage and coefficient are small [3]. As the temperature rises (between 75° and 130°), the dipole gets enough energy to move and the pyroelectric voltage at about 130°, is maximum due to depolarization of 90° domains in orthorhombic structure. When the temperature rises to about 193°, the 180° domains reorient and the rhombohedral phase become dominant.

In that group of specimens, the grain size was smaller and the change of direction of domains was simpler than the others, so the pyroelectric coefficient and the fluctuation be greater.

The grain size of pyroelectric ceramic $Pb(Ti_xZr_{1-x})O_3$ with $x = 0.07$ with increasing the sintering time, was increased. With increasing the grain size, possibilities for changing the direction of ferroelectric domains and fluctuation in pyroelectric coefficient become less. Study of SEM photographs show that the pyroelectric ceramics PZT have four types of ferroelectric domains with orientations of 71°, 90°, 109° and 180°.

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